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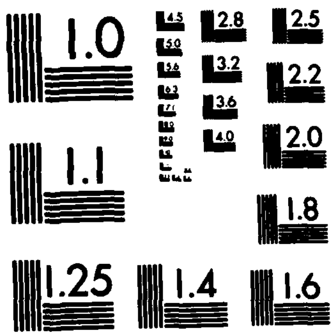
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DENSIFICATION OF PRECIPITATED YTTRIA STABILIZED ZIRCONIA (YSZ) TO ACHIEVE TRANSLUCENT PROPERTIES

by

R. C. Buchanan and D. M. Wilson

November 30, 1982

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20. Abstract

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Precipitated yttria (8.0 wt%) stabilized zirconia powders (YSZ) were sintered in the range 1150°C–1350°C using Al_2O_3 and B_2O_3 additions. A (2:1) Al_2O_3:B_2O_3 additive mixture at $\sim$ 2 vol% concentration, gave highest densities when sintered at 1200°C/1 hr. Washing of the powders to remove residual Cl was necessary to achieve high densification below 1300°C. Samples obtained were optically translucent at 1 mm thickness with average grain size 0.2–0.4 μm. Pore size increased with sintering temperature but numbers were significantly reduced. Densification by liquid phase sintering was evident with B_2O_3 present, but mechanisms for Al_2O_3 additions were not clearly established.

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Precipitated yttria (8.0 wt%) stabilized zirconia powders (YSZ) were sintered in the range 1150°C–1350°C using Al_2O_3 and B_2O_3 additions. A (2:1) Al_2O_3:B_2O_3 additive mixture at ~ 2 vol% concentration, gave highest densities when sintered at 1200°C/1 hr. Washing of the powders to remove residual Cl was necessary to achieve high densification below 1300°C. Samples obtained were optically translucent at 1 mm thickness with average grain size 0.2–0.4 µm. Pore size increased with sintering temperature but numbers were significantly reduced. Densification by liquid phase sintering was evident with B_2O_3 present, but mechanisms for Al_2O_3 additions were not clearly established.

Introduction

Yttria stabilized zirconia (YSZ) is used extensively as a solid electrolyte for oxygen sensor and fuel cell applications due to its high ionic conductivity.[1,2] The sintering temperatures for most zirconia powders are in the range of 1700-1800°. Using ultrafine, coprecipitated powders, nearly fully dense bodies can be obtained at 1350°-1400° with proper processing techniques.[3,4] A previous study[5] has shown that B_2O_3 and calcium borate additives are effective in bringing about full densification by liquid-phase assisted sintering of ultrafine grained calcia stabilized zirconia powders (CSZ) at 1200°. The borate additives were, however, ineffective in densifying YSZ at concentrations less than 20 vol%, due to insufficient wetting of the YSZ particle surfaces.[5] Significant fluxing of magnesite refractories at high temperature has also been observed with borate additives.[6]

Other sintering aids to YSZ have been found to be effective in varying degrees. Al_2O_3 additions (up to 1 wt%) to commercial YSZ when fired ≤ 1500° have been reported to increase sintered densities significantly.[7,8] These results implied that Al_2O_3 rich melts were formed with existing impurities in the YSZ and were effective in bringing about increased wetting and densification of the YSZ. An even more reactive melt could be expected to form from the simultaneous presence of Al_2O_3 and B_2O_3 in the additive phase to the YSZ.

In this study the Al_2O_3-B_2O_3 system was investigated as a sintering aid for ultrafine grained YSZ powders. The achievement of significant optical and IR translucency was anticipated in view of the effectiveness of borate melts in the sintering of CSZ.

Experimental

The powders used in this study were coprecipitated yttria (8.0 wt% ~ 6.6 mol) stabilized zirconia. Typical lot compositions and physical properties for the powders used are shown in Table 1.

The residual chlorine content of the YSZ powders in Table 1 averaged about 0.8 wt%. Presence of the Cl^- ions was found to decrease significantly the final densities achieved with the YSZ bodies. This was especially noticeable for the bodies sintered ≤ 1300°C. Washing of the coprecipitated powders to reduce the Cl^- ion content was carried out by subjecting a 1.0 vol% suspension of the YSZ powder in distilled water to ultrasonic vibrations for 15 minutes, followed by centifuging and decanting of the liquid. This procedure, which is similar to that used by Scott and Reed,[8] was repeated 3-4 times. Chemical analysis of the washed powders indicated a reduction in Cl content from 0.8 wt% to ≤ 0.04 wt%.

Reagent grade boric anhydride (B_2O_3) and fine grained reagent grade aluminum hydroxide ($Al(OH)_3 \cdot 3H_2O$), 99.9% pure, were added as sintering aids. The batches were milled for 12 hours with ZrO_2 balls in polyethylene jars using a 2:1 mixture of 2-propanol and deionized water. A binder mixture consisting of 1.0 wt% carbowax and 1.0 wt% PVA was then added to the jars and milled for an additional 1.5 hr. The milled suspensions were spray-dried** and pellets 1.6 cm diameter and ~ 0.15 cm thick were pressed, using uniaxial compaction pressures of 220 MPa. The pellets were fired on stabilized ZrO_2 setters in a $MoSi_2$ furnace in the range 1150°C-1350°C for up

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** Buche Laboratory Spray Dryer, Brinkman Instruments, New Jersey.

Table 1

Typical Lot Analysis for Yttria Stabilized Zirconia (YSZ) Powder*

Composition

Element	Wt%	Element	Wt%
ZrO_2	88.0	MgO	0.1
Y_2O_3	8.0	Fe_2O_3	0.01
HfO_2	1.6	H_2O	2.2
Al_2O_3	0.2	Cl	0.8
CaO	0.3	Na_2O	0.26
		SiO_2	0.1

Physical Properties

Stabilizer	Yttria, Y_2O_3
Crystalline Phase	Cubic
Crystallite Size	0.02-0.03 microns
Agglomerate Size	0.1 micron**
Surface Area (BET)	50 m^2/gm

*Zircar Corporation Powder Type ZYP, Florida, NY.

**Greater than 90% less than 0.1 micron when deflocculated in water acidified to pH 2.0 in HCl.

to 24 h soak time. Samples containing B_2O_3 were quenched from 800°C to avoid cracking, due to crystallization of a Y_2O_3-B_2O_3 phase at ~ 600°C.

Bulk densities of the samples were determined by water displacement technique. Theoretical densities (ρThD)were calculated using a series mixing formula, and known densities for the constituents as follows: YSZ (6.05 g/cm^3), boric oxide (2.46 g/cm^3) and alumina (3.98 g/cm^3). The formula used was:

$$\frac{1}{\rho ThD} = \frac{V_1}{\rho_1} + \frac{V_2}{\rho_2} + \frac{V_3}{\rho_3} \quad (1)$$

where V_1, V_2 and V_3 are the volume fractions of the respective phases with corresponding densities ρ_1, ρ_2, and ρ_3. Calculated theoretical densities for selected compositions used in this study are given in Table 2.

DTA data were obtained for all raw material constituents as well as for mixed powders of Al_2O_3 + B_2O_3, Y_2O_3 + B_2O_3 and pressed pellets of YSZ + Al_2O_3, YSZ + B_2O_3, and YSZ + Al_2O_3 + B_2O_3 mixtures. Additives to the YSZ powders (Al_2O_3 + B_2O_3) were generally less than 2.0 vol%, with a weight ratio in the range of 1:1 to 1:2. TGA weight loss measurements were carried out on some powders and pressed pellets up to 1100°C, and these were supplemented by weight loss measurements from the sintered samples up to 1250°C. A Dupont 1090 Thermoanalyzer System was used for the DTA and TGA measurements. Microstructures of fracture sections as well as polished and thermally etched sintered sections were examined using SEM and energy dispersive X-ray analysis (EDAX) techniques.

IR transmission spectra on polished samples 0.3-0.6 mm thick, were examined using a Nicolet FT-IR spectrophotometer.

Results

Figure 1 shows DTA heating and cooling curves for the Y_2O_3-B_2O_3 powder mixture (1:1 ratio). An apparent phase formation at 720°C and melt endotherms at 760° and 1120°C were observed on heating. On cooling, a sharp exotherm was obtained at 600°C. This latter can be attributed to crystallization of a Y_2O_3 containing phase from the borate rich melt phase existing above 750°. This crystallization caused cracking of YSZ samples sintered with B_2O_3 when cooled normally, possibly due to differential thermal expansion of the precipitated phase and Y_2O_3 segregation to the borate rich grain boundary phase. The cracking of the fired samples could readily be avoided by quenching the samples in air from ~ 800°C. It should be noted, moreover, that the amount of Y_2O_3 drawn from the cubic matrix grains was not sufficient to cause significant destabilization of the matrix, as can occur when B_2O_3 is added to calcia stabilized zirconia (CSZ),[5] or even magnesite refractories.[6]

The DTA traces for the other powder mixtures and pellets showed mainly the expected reactions and no crystallization peaks on cooling. TGA analysis up to 1100°C and weight loss data on the first samples up to 1350°C indicated a steady loss of chlorine, where present, from 900°C up to about 1300°C. Some loss of boron could also be inferred from the slightly greater weight losses observed for sintered samples containing B_2O_3.

The effect of Al_2O_3 additions on the density of YSZ is shown in Fig. 2. As noted in Table 1, 0.2 wt% Al_2O_3 was already present in the YSZ powders as an impurity. Al_2O_3 additions improved the fired densities significantly up to 0.65 wt% Al_2O_3, even at 1200°C. This improvement in densification can be attributed to development of a liquid phase from the Al_2O_3 additive and existing impurities such as MgO, CaO, and Na_2O. Such a CaO-MgO-Al_2O_3 eutectic melt phase does in fact exist at 1345°C.[10]

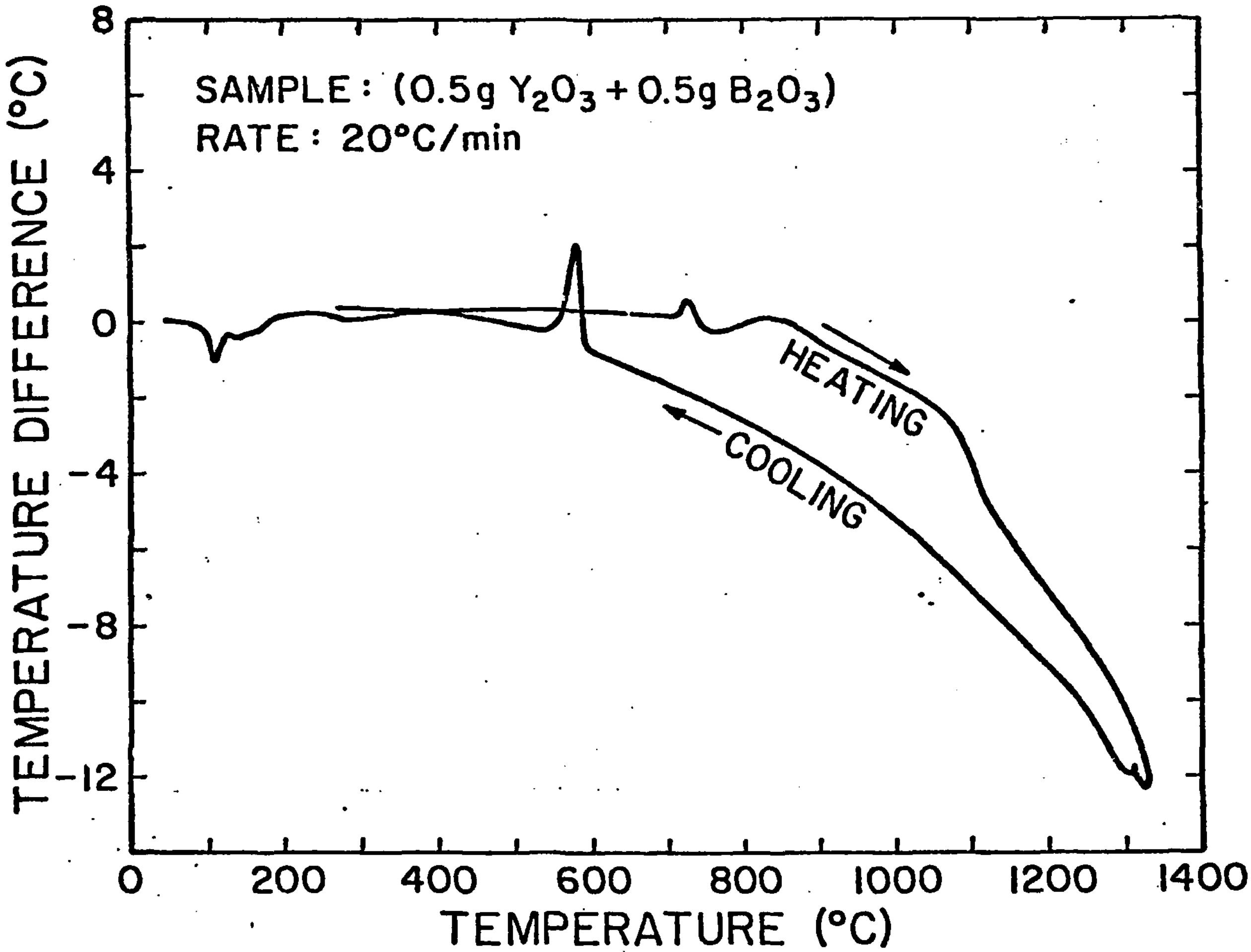


Fig. 1. DTA traces for (1:1) $Y_2O_23:B_2O_3$ sample showing heating reactions and phase formation (~ 600°C) on cooling.

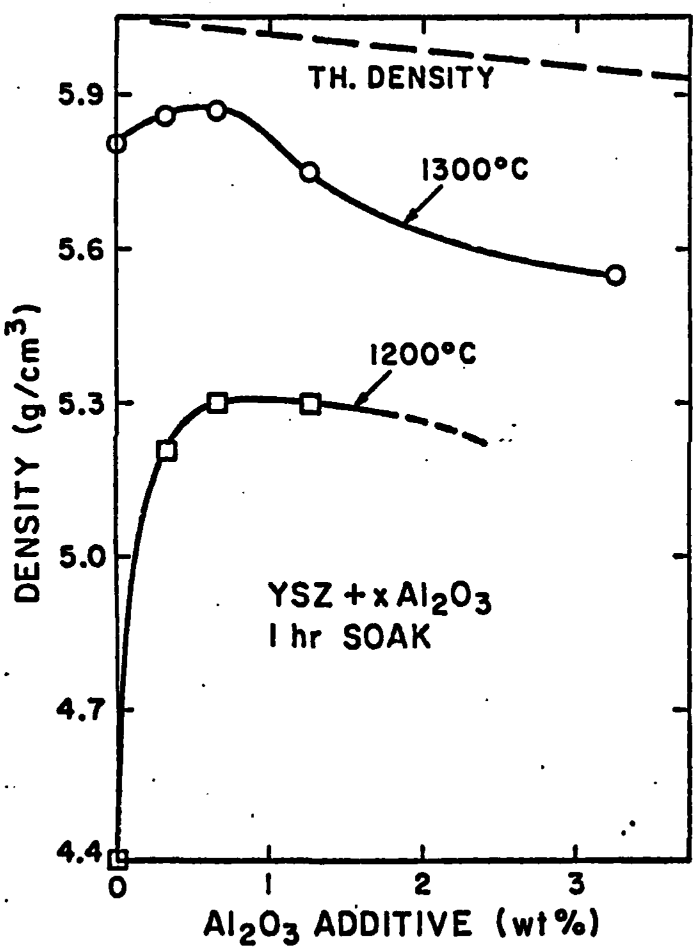


Fig. 2. Effect of Al_2O_3 addition on densification of YSZ at 1200°C and 1300°C/1 h.

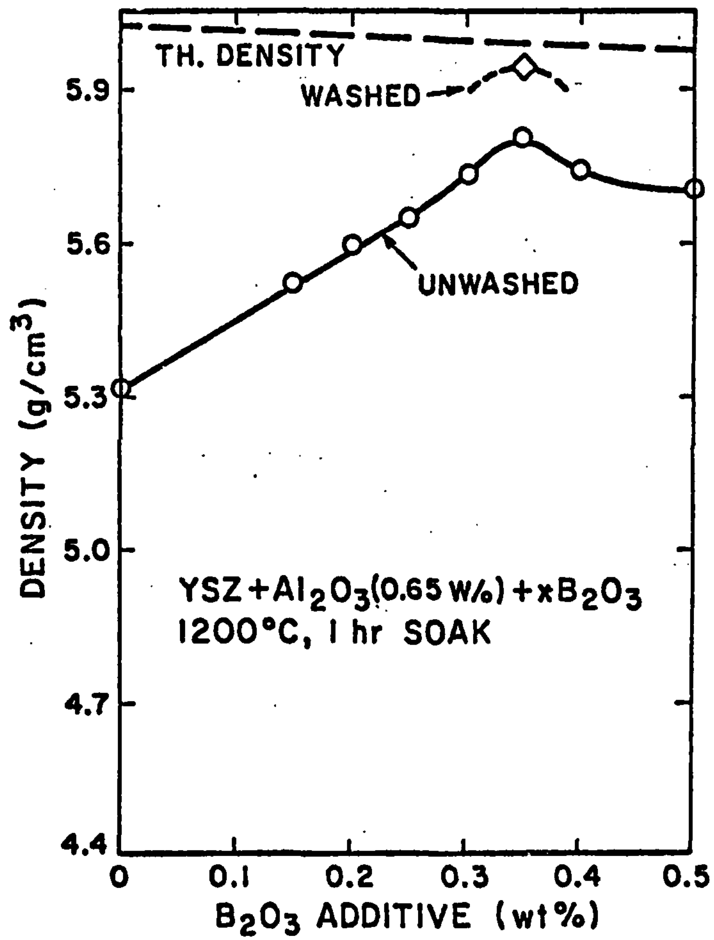


Fig. 3. Effect of B_2O_3 additions and processing on densification of YSZ + Al_2O_3 sample at 1200°C/1 h.

Additions of B_2O_3 to the YSZ-Al_2O_3 composition (99.35 wt% YSZ + 0.65 wt% Al_2O_3) with the highest density, further increased the density up to a B_2O_3 concentration of 0.35 wt% (Fig. 3). This optimal additive concentration of (Al_2O_3 + B_2O_3) yielded the highest density in the YSZ-Al_2O_3-B_2O_3 system (5.94 = 99.2% Th.D at 1200°/1 hr) when washed YSZ powder was used.

As was shown by Scott and Reed,[9] chlorine present in coprecipitated YSZ can retard densification by 150° or more. This is borne out by Fig. 4. Washed YSZ + Al_2O_3 + B_2O_3 powders (D) reached nearly full density at 1200° in 1/2-hour, while an identical composition (C) using an unwashed YSZ powder achieved similar density (5.93 g/cm^3) only at 1275°/4 h (Fig. 5). The batch compositions for the samples in Figs. 4 and 5 correspond to those given in Table 2.

Figure 5 also shows a marked decrease in density with sintering temperature above 1200°C (4 h soak) for the washed borate containing sample. For the unwashed sample some decrease is evident also above 1250°C. In contrast, both the YSZ and (YSZ + Al_2O_3) samples showed continuous increases in density with temperature, finally a achieving higher density at 1350°C.

The densification behavior as a function of soak time at 1200°C and 1275°C for the YSZ and modified samples is shown in Figs. 6a and 6b. For modified YSZ powders the densification rate was rapid below 2 h soak but gradually decreased with time up to 10 h. Between 10 and 24 h soak time, the densities actually decreased slightly. This data is given in Table 3 which shows the final densities achieved for selected compositions.

Table 2

Calculated Densities for Selected Compositions in ($YSZ + Al_2O_3 + B_2O_3$) System

Sample Number	Composition	Calculated Th.D. (g/cm^3)
A*	YSZ (8 wt% Y_2O_3)	6.05
B	YSZ + 0.65 wt% Al_2O_3	6.03
C	YSZ + 0.65 wt% Al_2O_3 + 0.35 wt% B_2O_3	5.98
D	Composition C (washed)	5.98

*Note that all compositions contained ~ 0.2 wt% Al_2O_3 as base impurities.

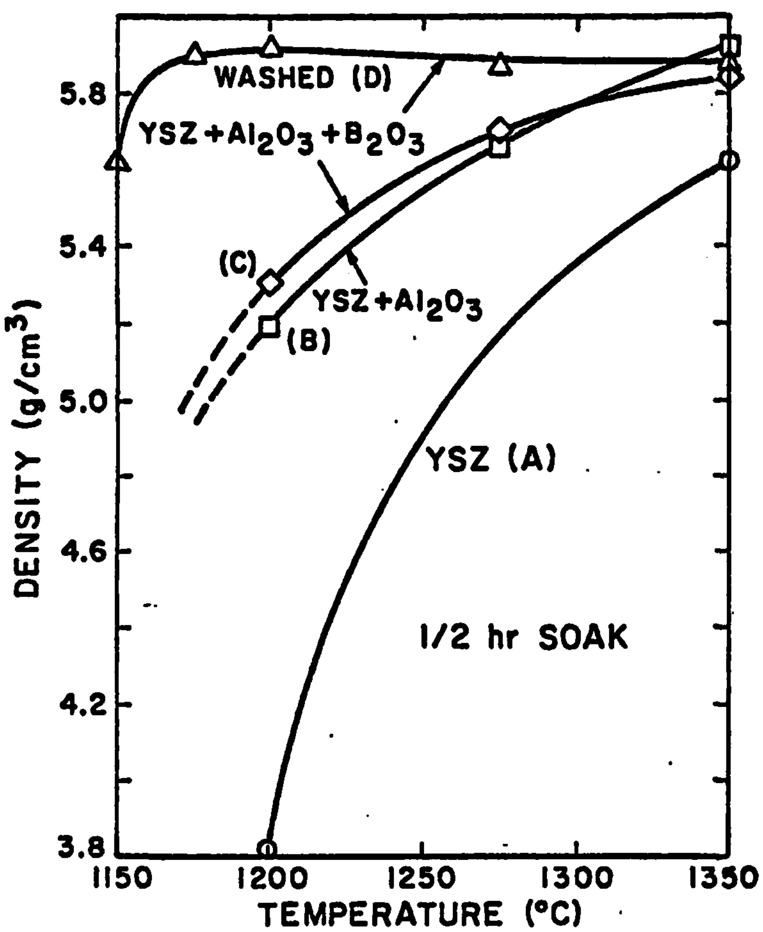


Fig. 4. Effect of temperature (1/2 h soak) processing and composition on densification of YSZ powders.

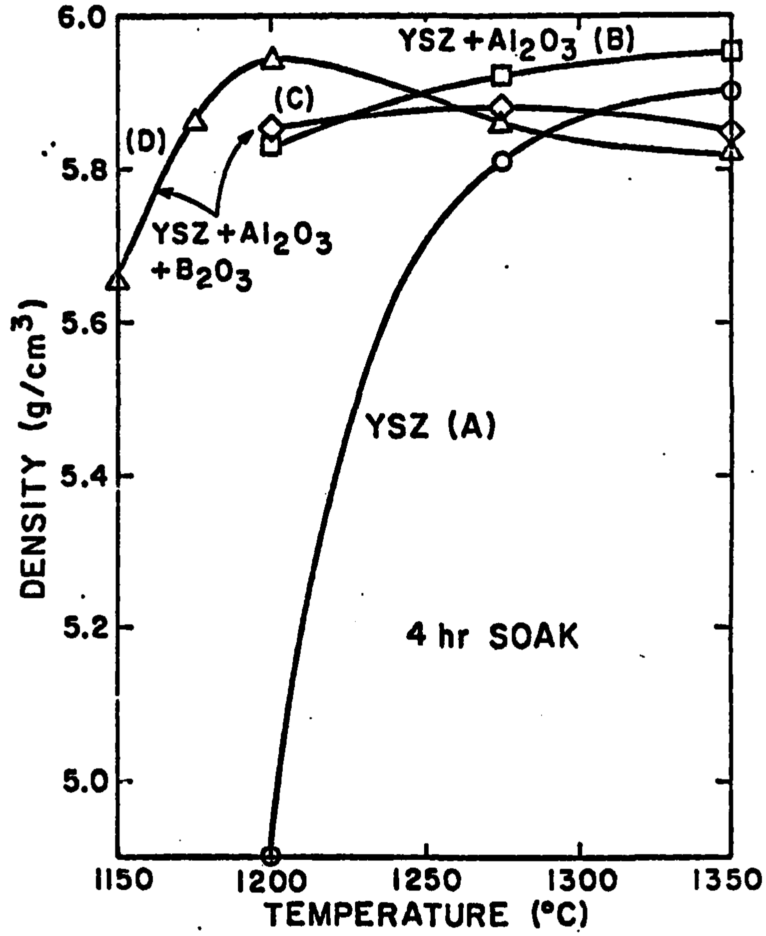


Fig. 5. Comparison of densification of YSZ in Fig. 3 for 4 h soak time at temperature

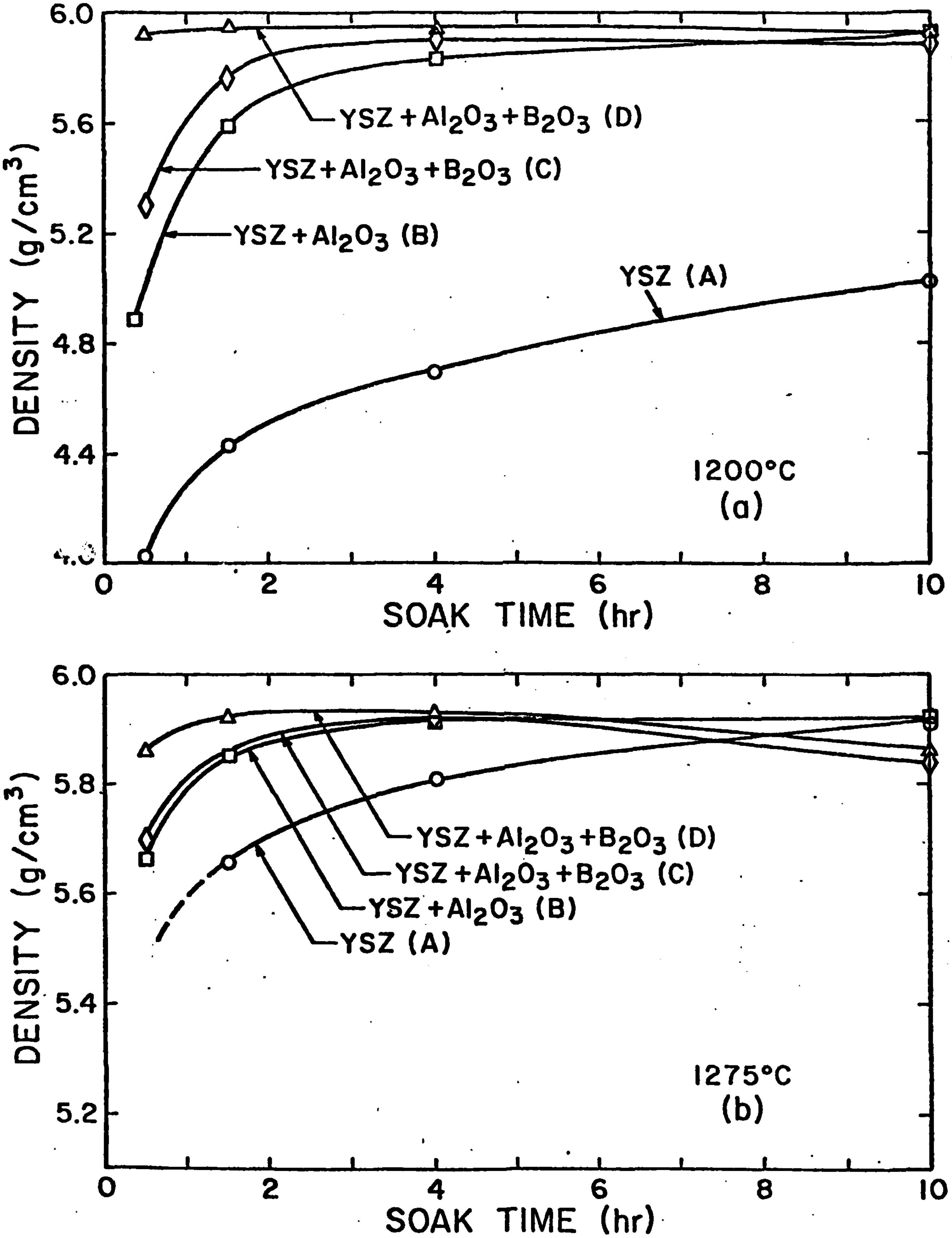


Fig. 6. Effect of soak time and additive composition on densification of YSZ at (a) 1200°C and (b) 1275°C.

From Table 3 the maximum densities achieved were at 1200°C for samples C, D, and E for soak times ≥ 4 h. For sample C this maximum was at 1275°C/4 h, reflecting the higher Cl content. All samples showed increased densification with temperature for short soak times (~ 0.5 h), but densities generally declined for soak times ≥ 4 h at temperatures above 1200°C, except for the YSZ samples which showed a monotonic density increase with temperature and soak time. Reasons for the decrease in density of YSZ + additive samples at higher temperatures and longer soak times are not evident. This phenomenon was, however, observed by Bernard[8] with Al_2O_3, Fe_2O_3, and TiO_2 additives to YSZ. A likely explanation is slight destabilization of the YSZ structure[5] and the creation of porosity by volatile constituents (Cl, B_2O_3) on being eliminated from the ceramic.

Translucency of the samples was not, however, reduced by the higher temperature firing, and was in some cases enhanced. This condition reflects the elimination of small pores from the sintered bodies and a corresponding growth of a few large pores. Polished samples 1 mm thick showed a high degree of translucency when examined optically. In contrast, IR transmission was below 20% but this partly reflects difficulties in measurement including surface reflections.

Pore volume distribution was confirmed by SEM analysis of the fracture and polished sections of the sintered samples. Figures 7a and 7b show polished and thermally etched sections of sample D (washed YSZ with 1.0 wt% (Al_2O_3 + B_2O_3). Grain sizes were in general less than 0.2 μm on average. Small, uniformly distributed pores were observed for the 1200°C samples.

At 1350°C, significant pore growth and coalescence appeared to have taken place, since relatively few, but larger pores were observed. Average grain size was slightly larger at about 0.3 μm. Mechanisms for the essentially complete densification of YSZ samples as low as 1200°C, with Al_2O_3 and B_2O_3 additions, have not been satisfactorily established. Elimination of the Cl (900°C–1300°C) during sintering of the ceramic would likely have the dual effect of inhibiting mass transport and creating porosity, both inimical to good densification. Inhibition of densification, where Cl is present in the precursor powders may, therefore, be attributed to these factors.

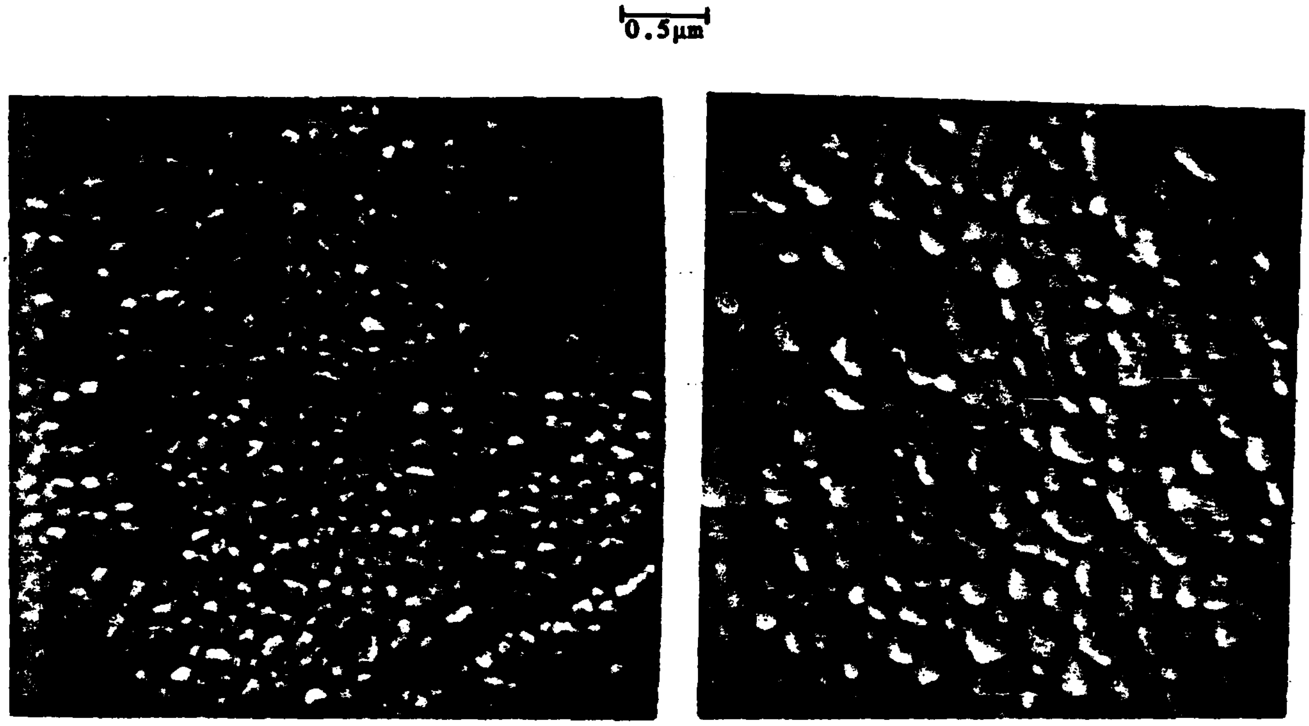
With Al_2O_3, densification appears to peak at about 0.6 wt%. This is in agreement with work by Bernard,[8] which showed a similar temperature and composition dependence for Al_2O_3 additions to precipitated YSZ powders. Little evidence of liquid phase was found in the resultant microstructures, hence the Al_2O_3 was considered as a dopant which enhanced sintering possibly by grain boundary activation.[8] A somewhat analagous explanation has been proposed by Butler and Drennen,[11] with Al_2O_3 acting as a scavenger for SiO_2 impurities at 1600°C, removing it from grain boundary locations and thus enhancing densification. In the present study, it is believed that liquid phase formed by Al_2O_3 with MgO, CaO, and SiO_2 impurities provide a more reasonable explanation for the experimental data. The fact that a similar peak and densification behavior is observed with B_2O_3 present, where the presence of liquid phase has clearly been established, lends credence to this hypothesis. More detailed TEM and analytical work is, however, clearly indicated.

Table 3

Fired Densities for (YSZ + Additive Samples) at Different Soak Temperatures and Times

Sample	Soak Time (h)	Fired Densities (Bulk--g/cm^3)* 1200° Bulk	1200° %Th.D.	1275° Bulk	1275° %Th.D.	1350° Bulk	1350° %Th.D.
A	0.5	4.02	66.4	5.4	89.3	5.63	93.1
	4.0	4.69	77.5	5.81	96.0	5.90	97.5
[YSZ(8 wt% Y_2O_3)]	24.0	5.25	86.8	5.91	97.7	5.96	98.5
B	0.5	4.90	81.3	5.82	96.5	5.88	97.7
	4.0	5.82	96.5	5.91	98.0	5.89	97.8
[YSZ + 0.65 wt% Al_2O_3]	24.0	5.95	98.7	5.89	97.8	5.88	97.7
C	0.5	5.3	88.6	5.70	95.2	5.85	97.7
[YSZ + 0.65 wt% Al_2O_3	4.0	5.85	97.7	5.93	99.1	5.82	97.2
+ 0.35 wt% B_2O_3]	24.0	5.90	98.6	5.81	97.1	5.82	97.2
D	0.5	5.92	98.9	5.86	97.9	5.88	98.2
[YSZ + 0.65 wt% Al_2O_3	4.0	5.94	99.2	5.90	98.6	5.77	96.4
+ 0.35 wt% B_2O_3 (Washed)]	24.0	5.90	98.6	5.84	97.6	5.77	96.4
E	0.5	4.90	81.1	5.66	93.0	5.91	97.9
[YSZ + 0.325 wt% Al_2O_3]	4.0	5.83	96.5	5.92	98.0	5.90	97.7
	24.0	5.98	99.0	5.90	97.8	5.88	97.4

*± 0.01 g/cm^3.



(a) 23,000X

(b) 23,000X

Fig. 7. SEM photomicrographs of polished and thermally etched sections of YSZ + (2:1) [Al_2O_3:B_2O_3] samples sintered at (a) 1200°C/1 h and (b) 1350°C/4 h.

Conclusion

1. Sintering studies carried out on precipitated YSZ powders with Al_2O_3 and B_2O_3 additions showed significant enhancement in densification in the range 1150°C-1300°C.

2. Highest densities (> 99.2% ThD) were achieved with washed powders (to remove Cl) at 1200°C/1 hr with a ~ 2 vol% (2:1) Al_2O_3:B_2O_3 additive mixture. Presence of the Cl was found to inhibit densification below 1300°C.

3. Dense samples were optically translucent with average grain size 0.2-0.4 μm.

4. Mechanisms for the accelerated densification have not been clearly established.

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Summary of Work Accomplished Under Contract US NAVY-N-00014-80-K-0969

1. Reports

Report issued under this contract include the following:

a. R. C. Buchanan and S. Pope, "Optical and Electical Properties of Yttria Stabilized Zirconia (YSZ) Crystals," (ONR Report 5), University of Illinois, Urbana, IL (September 1981).

b. R. C. Buchanan and J. Boy, "Effect of Coprecipitation Parameters on Powder Characteristics and On Densification of PZT Ceramics," (ONR Report 6), University of Illinois, Urbana, IL (September 1982).

c. R. C. Buchanan and D. M. Wilson, "Densification of Precipitated Yttria Stabilized Zirconia (YSZ) to Achieve Translucent Properties." (ONR Report 7), University of Illinois, Urbana, IL (November 1982).

3. Papers

a. R. C. Buchanan and S. Pope, "Optical and Electical Properties of Yttria Stabilized Zirconia (YSZ) Crystals," Accepted, J. of Am. Ceram. Soc., 1982.

b. R. C. Buchanan and J. Boy, "Effect of Coprecipitation Parameters on Powder Characteristics and On Densification of PZT Ceramics," submitted to J. of Am. Ceram. Soc., 1982.